

# **The magnetic properties of aggregate polycrystalline diamond: Implications for carbonado petrogenesis**

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## **Popular Summary**

Carbonados are a type of diamond, which are made up of many aggregates of small crystalline diamonds or microdiamonds. The term "carbonado" comes from the Portuguese word carbonated. They are only found in sedimentary deposits in the Central African Republic (CAR) and the Bahia Province of Brazil. They were once the source of the world's supply of industrial diamonds. Their origin is uncertain but several mutually exclusive hypotheses have been proposed. These theories are: (1) extraterrestrial, that is they formed from the dust cloud of original solar nebulae; (2) produced by the high temperatures and pressures of the Earth's mantle; (3) or as the result of an extra-terrestrial impact into a carbon rich layer of sediment. Our study was done to further the understanding of their origin. We measured the magnetic properties on some twenty samples from the CAR. An earlier study was done on whole samples of carbonados and the "common" or kimberlitic diamond. Our work differed in that we started at the surface and subsequently removed the surface layers (by days of acid immersion) into the interior; measuring the magnetic properties at each interval. This procedure permits us to monitor the distribution of magnetic substances within the samples. Our results showed that the magnetic carriers are distributed on the surface including the open pores and that the carbonado interior is essentially non-magnetic. This result suggests that the initial formation environment was deficient in magnetic particles. Such a situation could indicate that their formation was the result of an extra-terrestrial body impacting carbon-rich sediment. Obviously, more work will be required on isotopic and chemical analyses before a more detailed origin can be determined.



## **The magnetic properties of aggregate polycrystalline diamond:**

### **Implications for carbonado petrogenesis**

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#### **Abstract**

Carbonados are aggregate polycrystalline diamonds. They are found in placer deposits of the Central African Republic (CAR) and the Bahia Province of Brazil. Their origin is uncertain but several mutually exclusive hypotheses have been proposed ranging from extraterrestrial to crustal, and mantle provenance. In an endeavor to further our understanding of these diamonds, we performed a series of magnetic characterization studies on some twenty samples from the CAR.

Our results reveal material with contrasting magnetic hysteresis behavior and magnetization. Acid leaching of carbonados permits us to monitor the distribution of magnetic carriers within the samples. An absence of grain-size dependence on magnetization loss indicates that the magnetic carriers are distributed at the vitreous surface including open pores and that the carbonado interior is essentially devoid of them. The non-magnetic interior of carbonados suggests that the initial formation environment was deficient in magnetic particles.

The formation of the magnetic carriers is thus closely linked with the origin of the smooth surface, perhaps during the reentry of carbonados into the Earth's atmosphere. Such an event followed their formation possibly as a result of a bolide impacting carbon-rich sediment. We do not exclude the idea that the extraterrestrial bolide may have been rich in C-bearing species such as diamond and polycyclic aromatic hydrocarbons (PAHs). Partial ablation of the bolide during the entry into the Earth's primordial, oxygen-poor atmosphere resulted in a fusion of the originally porous material and a decrease in the



original pore density at the carbonados' surface. Secondary mineralization of the impact-generated melt and/or the products of later diagenetic processes filled the remainder of the open pores.

### **Introduction:**

Carbonados are sintered polycrystalline microdiamond aggregates with a porous, ceramic texture and a melt-like surface patina [1, 2]. The closed pores contain significant (up to 0.3 wt. %) amounts of the total content of organic material [3]. They lack significant mantle mineral inclusions and are distinct from kimberlitic diamonds [1, 4, 5]. They have only been found in abundance in alluvial deposits in Bahia, Brazil and the Central African Republic (CAR). These two localities may constitute a single source because they both lie on the West Congo-Salvador craton, only about 1500 km apart in a plate reconstruction of Gondwanaland [6]. These deposits are ~1.5 Ga old while the carbonados are between 2.6 – 3.8 Ga [7]. Random orientation of the crystallites increases the structural integrity of the material with respect to the larger, single diamond crystals, which are subject to disintegration along cleavage planes. Precisely how the carbonado microcrystals fused together and what mechanism caused the high porosity with trapped PAHs is poorly understood, doubtless due to fact that the origin of the carbonados themselves is unclear.

Several carbonado genesis theories have been proposed [2]. A crustal origin is favored by some and is supported by the following isotopic and mineralogical data. Carbonados have light Carbon (C) and Helium (He) isotopic signatures [8, 9], contain an enrichment of light rare-earth elements (REEs) [8, 10] and have significant amounts of atmospheric noble gases [11]. They contain significant amounts of He, which appears to be inconsistent with the expected depletion of this element at mantle temperatures [4, 12]. Carbonados also have high porosity, which is incompatible with high-pressure mantle

conditions [4]. These pores are filled with polycyclic aromatic hydrocarbons (PAHs) [3, 13, 5].

An irradiation origin has been proposed [11, 14] based on the presence of high concentration of parentless fission products (Xe, Kr) in both Brazilian and African carbonados. The rationale is that large amounts of fissiogenic species require vicinity of finely dispersed U-rich material over geological time scales. The radiation associated with this material then contributes to the diamond formation [11].

An alternative mantle origin was proposed based on the similarity of the REE pattern in kimberlites and carbonados [15, 16], the presence of Nitrogen (N) platelets in the microcrystalline diamond (recorded by IR spectroscopy [15], [17]) and low concentration of syngenetic inclusions of rutile, ilmenite, and magnetite in the carbonado aggregate. All of these characteristics are consistent with the high pressure and temperature conditions expected from a mantle origin [18].

A further hypothesis proposed that carbonados formed as a result of a primordial extraterrestrial bolide, impacting carbon-rich crustal sediments [19, 20, 21]. This is suggested by the rare and controversial occurrence (only in the Yakutian carbonado [19, 22]) of the hexagonal diamond polymorph, lonsdaleite, frequently found in high-pressure diamonds from several meteorite impact sites [20], and by the observation of planar deformation features and characteristic isotopic signatures, suggestive of a shock history [21].

Finally, Haggerty proposed that carbonados are of extraterrestrial origin [4], based on the following evidence: the high abundance of C in the solar system; the existence of diamonds in primitive meteorites; the overlapping range of C isotopic composition with meteoritic nanodiamonds; the high concentrations of planar deformation features consistent with shock metamorphism; the radiation damage possibly due to cosmic ray exposure; the high porosity suggestive of loss of H gained by solar implantation; the age (2.6-3.8 GA); the restricted distribution of carbonado; and their melt like surfaces.

This report focuses on our investigation of the magnetic properties of carbonados and the nature of their magnetic components. Carbonados are commonly inter-grown with hematite, magnetite, ilmenite, and chromite [2, 18, 23, 24]. In addition the diamond matrix contains small amount of the unusual particles  $\alpha$ -Fe, Ni, Cr, Fe-Mn, Ti, SiC, taenite  $\gamma$ -(Fe, Ni) [18, 21], thought to be related to an early reducing stage of carbonado history.  $\alpha$ -Fe and taenite are the most abundant among these compounds [18]. All of the other inclusions are thought to be of secondary origin [18].

Collinson [25] conducted a study of the magnetic properties of polycrystalline diamonds including carbonados from Brazil and the CAR. He reported that, in comparison to other diamonds, carbonados have very little magnetic material resulting in low magnetic intensities. The NRM (natural remanent magnetization) intensity  $(1-10) \times 10^{-5} \text{ Am}^2\text{kg}^{-1}$  was stable with a very small decrease ( $\sim 10\%$ ) after demagnetization to 100 mT. These NRM directions showed a steady migration to stable primary end-points with irregular intensity decay curves. Conventional thermomagnetic experiments utilized to determine Curie points indicate a discontinuity at 120–160 °C for some carbonados suggesting a Curie point which has not been identified with a mineral species [25]. The initial magnetic susceptibility was in the range  $\sim 5 \times 10^{-8} \text{ m}^3\text{kg}^{-1}$ . During isothermal remanent magnetization (IRM) acquisition, there were no indications of saturation up to an applied field of 800mT. The REM ratio (NRM/saturation isothermal remanent magnetization) is 0.1-0.01 as opposed to a range of terrestrial material, which is typically 0.01-0.001 [26].

### **Materials and methods:**

We recently obtained twenty carbonados from the CAR. They are of variable size, color and morphology (Figure 1). These samples possess diverse pore sizes and concentration, with both decreasing on glassy smooth surfaces (e. g. D10, D11, and D12, Figure 1). All samples were subjected to ultrasound cleaning before measurement. NRM and Saturation Isothermal Remanent Magnetization (SIRM) were measured with a Supercon-

ducting Rock Magnetometer, (SRM, Superconducting Technology). Hysteresis properties of strongly magnetic samples were measured with a vibrating sample magnetometer (VSM, Lake Shore *model 7300*). Magnetic fields of up to 2T were supplied via a large, water-cooled, 12-inch Varian magnet, driven by a Tidewater bipolar power supply. All samples were further examined by optical and scanning electron microscopy (SEM).

Samples with large magnetic signatures were soaked in a 50:50 mixture of 50 wt.% HF and 6N HNO<sub>3</sub> for 24 hours (h) to remove surface contamination. Samples were then further soaked in 6N HCl, which was replenished every 72 h. These samples were also cleaned ultrasonically in the course of the acid change. Mass and saturation isothermal remanent magnetization (SIRM) were measured after 14, 46, 64 days. After 64 days, samples continued to dissolve in HCl as indicated by the yellow coloration of the acid towards the end of the 72 h cycle. However, at this stage, the magnetic signature was found to have dropped significantly.

## Results

Figure 2a compares measurements of the bulk magnetic properties of our samples with the earlier work by Collinson [25]. Acid cleaning resulted in both loss of mass (Figure 2a) and saturation remanence (Figure 2b). A larger proportion of the magnetic signal was lost compared to the fractional mass loss. The extent of the mass loss (1-5%) was dependent on the sample's original mass (Figure 3a). However, the magnetization loss (60 – 99 % of initial SIRM) was essentially independent of the mass (Figure 3b).

Continuous acid treatment produced contrasting magnetic behavior in different samples. The treatment resulted in magnetic hardening (coercivity increases) in D-11 and D-15 (Figure 4). Samples D-3 and D-6 show constant resistance to the AF (alternating magnetic field) demagnetization during all dissolution steps (Figure 5). Acid treatment of samples D-12, D-13, D-14 and D-20 left magnetization carriers that had softer resistance against AF field than before treatment (Figure 6).



Table 1 summarizes the magnetic characteristics of our samples before acid treatment. NRM values were  $(0.1-10) \times 10^{-5} \text{ Am}^2\text{kg}^{-1}$ . This is consistent with measurements by Collinson [25] who reported the same range of NRM values (see Figure 2a). Saturation magnetization was in the range  $(1.0-500) \times 10^{-5} \text{ Am}^2\text{kg}^{-1}$  and REM ratios were 0.001-0.3. These hysteresis parameters were measured only on a limited number of samples due to the lower sensitivity of the VSM. Hysteresis loops were often constricted, indicating the presence of two magnetic components with different coercivities (see Figures 7b, c). One loop (sample D-15) resembled native iron based on the large saturation field (see Figure 7d). All of the samples displayed a diamagnetic component  $(-2 \times 10^{-2} \text{ A m}^2 \text{ kg}^{-1})$  owing to the presence of the diamond matrix.

### Discussion:

The most significant results we report are the independence of the magnetic properties on the mass of carbonados and the observation that most of the magnetization is lost following acid treatment. Thus, most of the magnetic carriers must be located on the surface and exposed to the acid. Since we ultrasonically cleaned these samples before etching in order to avoid surface contamination, magnetic carriers may be part of the original, smooth surface and exposed pores of the carbonado. However, their interior, which is not exposed to the acid and is therefore unrelated to their smooth surface, is relatively free of magnetic carriers. According to our detection limit for hysteresis parameters, the mass concentration of Fe particles (saturation magnetization =  $218 \text{ Am}^2\text{kg}^{-1}$ ) must be less than  $10^{-5} \text{ wt. \%}$ . Thus, there may still be room for a limited concentration of small inclusions of metallic components within the carbonado matrix [18, 21], but not much.

Our results suggest that the initial carbonado-forming episode must have taken place in an environment relatively free of magnetic materials. This event included the process of fusing the individual diamond subcrystals into an aggregate and the formation

of their bulk porous matrix. Apart from a possible metallic (Fe) content of up to  $10^{-5}$  wt. %, the absence of internal magnetic materials is inconsistent with a mantle origin [15, 16]. Such a model necessitates that crustal organic matter, commonly associated with magnetic oxides, could have been subducted into the mantle and converted to diamond, thereby explaining the light C isotopic signatures and high concentration of polycyclic aromatic hydrocarbon species present [15, 27]. If carbonados were formed in the mantle in keeping with this model, one would expect them to be found in the vicinities of ancient subduction zones. Carbonados however, *in sensu stricto*, have yet to be recovered from outside Brazil and the CAR [23, 24]. Furthermore, the high porosity exhibited by carbonados is difficult to interpret if they were formed under conventional high-pressure mantle conditions. Indeed, laser-induced luminescence indicates formation temperatures  $< 400$  °C [15, 28].

The radiogenic fission origin requires that U and Th in carbonaceous matter formed at least the initial “seeds” for the growth of the carbonados, in an environment free of magnetic carriers, under low-pressure conditions [7, 11, 29]. However, in order to continue the diamond growth further, another diamond forming process (perhaps meteorite impact) must be invoked due to the inefficient mass transport mechanism associated with carbonado formation by irradiation [29].

Meteorite impacts in C-rich sediment have been proposed as another mechanism for carbonado formation [20, 30]. During such impacts, organic matter or graphite nodules can be converted to diamond by shock metamorphism. If microdiamonds were part of the original meteorite, they might serve as useful nucleation centers [31]. The impact hypothesis can not only explain the light C isotope ratios but also the common inclusions of crustal minerals within the carbonado pores. Our magnetic data further constrain that the organic matter was free of magnetic carriers prior to such an impact. Tektites are formed by a bolide impacting a silica-rich crust and commonly contain significant amounts of magnetic material [32]. Organic matter, however, can cumulate significant

amounts of carbon with miniscule amounts of magnetic carriers. Even though lonsdaleite (the hexagonal carbon polymorph commonly associated with impact diamonds) has not been reported in carbonados from CAR and Brazil, we stress that the impact model is still a viable mechanism. In addition, the microdiamonds are associated with several other known impact sites (e. g. Canyon Diablo, Popogai and Ries craters), but their dimensions are rather smaller than carbonado [33 - 39].

One important characteristic of carbonado is the abundance of pores. These pores contain up to 0.3 wt. % of organic matter [13]. The extracted C in this organic fraction is isotopically heavier ( $\sim$ -25.2 per mil) compared to C remaining as diamond matrix after extraction ( $<$ -28 per mil) and contains PAHs up to 0.002-0.004 wt. % of carbonado [5]. Thus, organic compounds other than PAHs dominate the organic content of carbonados. However, because the fraction of PAHs increases with decreasing grain size of crushed carbonado powder [5], it is inferred that these aromatic compounds were primarily resident within the closed pores of carbonados. The mean size of the diamond powder was much larger than that of the crystallites making up the carbonado and hence the actual content of PAHs in carbonado is likely to exceed the observed 0.002-0.004 wt.% [5].

We propose that at least three distinct phases were involved in the formation of carbonados. **Phase one** was the formation of the polycyclic aromatic hydrocarbons. **Phase two** was the nucleation of the isolated euhedral crystals ( $\sim$ 1 $\mu$ m), with incorporation of an abundant noble gas content. These crystals form the interior of carbonados with the abundant pore space (Figure 8a) that trapped PAHs. Both of these phases occurred in the relative absence of magnetic carriers with the exception of metallic impurities, which did not exceed  $10^{-5}$  %. **Phase three** was the formation of the vitreous, smooth surface (see Figure 8b) which resembles a fusion crust. The mechanism for this surface genesis is unknown but is most likely related to the formation of most of the magnetic particles within the exposed pores (Figure 9 a,b). These particles are responsible for the magnetic

signature of carbonados and are related to secondary precipitation during their residence time in the bedrock.

We thus associate formation of the majority of the magnetic carriers with the origin of the smooth surface. This may have occurred during the residence time in the Earth's crust via erosional forces associated with formation of crustal magnetic material within the carbonado pores. An alternative scenario is that the smooth, glassy patina is a fusion crust, formed perhaps during the reentry of carbonados into the Earth's atmosphere following their formation during the impact of a meteorite into carbon rich sediment. Carbonados partially ablated during entry into the Earth's early, O-poor atmosphere. This partial ablation resulted in a fusion of the originally porous material and a decrease in the original pore density. The remainder of the pores was filled by secondary mineralization of the impact-generated melt and/or later diagenetic processes. This process necessitates the accumulation of pure C in a form of organic-rich sediment early in the Earth history (2.6-3.8 BA).

We also can not rule out the possibility that carbonados could be the impactor itself [4] and could have formed in an interstellar environment perhaps in the wake of a supernova explosion. In this case, the formation of PAHs inside the pore space needs to be placed in an interstellar environment as well. However, magnetic carriers are formed in both cases after the smooth surface is formed most likely in a terrestrial environment.

The etching-dependent magnetic signature thus reveals part of the carbonado post-formational history. With respect to the hysteresis loops (Figure 7), it is clear that the nature of magnetic particles is bimodal and represents the combination of hard and soft magnetic material. This often results in constricted hysteresis loops. The resistance of magnetic remanence to an alternating magnetic field is commonly large (Figures 4, 5, and 6). The effect of etching on the magnetic resistance against AF demagnetization field is rather variable and echoes the oxide origin history of individual carbonados. Two carbonados (D-11 and D-15) experienced magnetic hardening upon acid treatment (Figure

4). These are the samples that lost more than 95% of their original saturation remanence. For these carbonados, the harder magnetic component is likely to be more primitive and indicates at least two precipitation phases for the magnetic carries. Samples D-3 and D-6 retained constant magnetic resistance during the acid treatment suggesting that their magnetic mineralogy does not change in the course of etching. This points to the genesis of a single magnetic phase. Samples with the dual coercivities and constricted hysteresis loops (D-12, D-13, D-14, and D-20) became magnetically softer following the acid treatment indicating that the softer component is more primitive in these samples and that the harder component was introduced later in the carbonados' history.

### **Conclusions:**

(i) The independence of magnetization on carbonado mass and the fact that a significant amount of this magnetization was lost during the acid treatment indicate that most of the magnetic material is located in pores with direct access to the sample surface.

(ii) The formation of the major magnetic component is either contemporaneous with that of the surface or the result of a later event.

(iii) The saturation-remanence dependence on the etching indicates that the magnetic properties have more- and less-primitive magnetic components with distinct coercivities. This likely relates to the environmental history of individual carbonado grains during their residence period in the terrestrial crust.

(iv) Our findings are consistent with carbonado formation associated with a meteorite impact where carbon rich matter is either the "target" or the "bullet".

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Table 1: Bulk magnetic properties of carbonados

sample	weight [mg]	NRMx10 <sup>-6</sup> [Am <sup>2</sup> kg <sup>-1</sup> ]	SIRM x10 <sup>-6</sup> [Am <sup>2</sup> kg <sup>-1</sup> ]	REM NRM/SIRM	Hc [mT]	J <sub>s</sub> [Am <sup>2</sup> kg <sup>-1</sup> ]
D1	238.2	4	101	0.0431	20	0.0002
D2	143.1	11	107	0.0991		0.0002
D3	233.3	5	524	0.0086		
D5	214.6	5	39	0.1367		0.0001
D4	160.5	99	393	0.2522	1.5	0.0057
D6	170.1	8	1067	0.0075		
D7	255.0	4	352	0.0109		
D8	197.7	7	20	0.3304		
D9	215.6	4	159	0.0222	50	0.0004
D10	175.1	6	1067	0.0061		
D11	5123.4	4	5607	0.0007	31	0.0105
D12	3456.8	3	200	0.0137	100	0.0002
D13	618.8	2	230	0.0087		
D14	467.4	2	736	0.0031	100	0.0015
D15	410.2	49	542	0.0911	0.5	0.012
D16	616.8	2	154	0.0143		
D17	519.9	2	104	0.0227		
D18	297.9	3	11	0.2940		
D19	405.0	2	151	0.0165		
D20	394.8	20	183	0.1080		0.0003

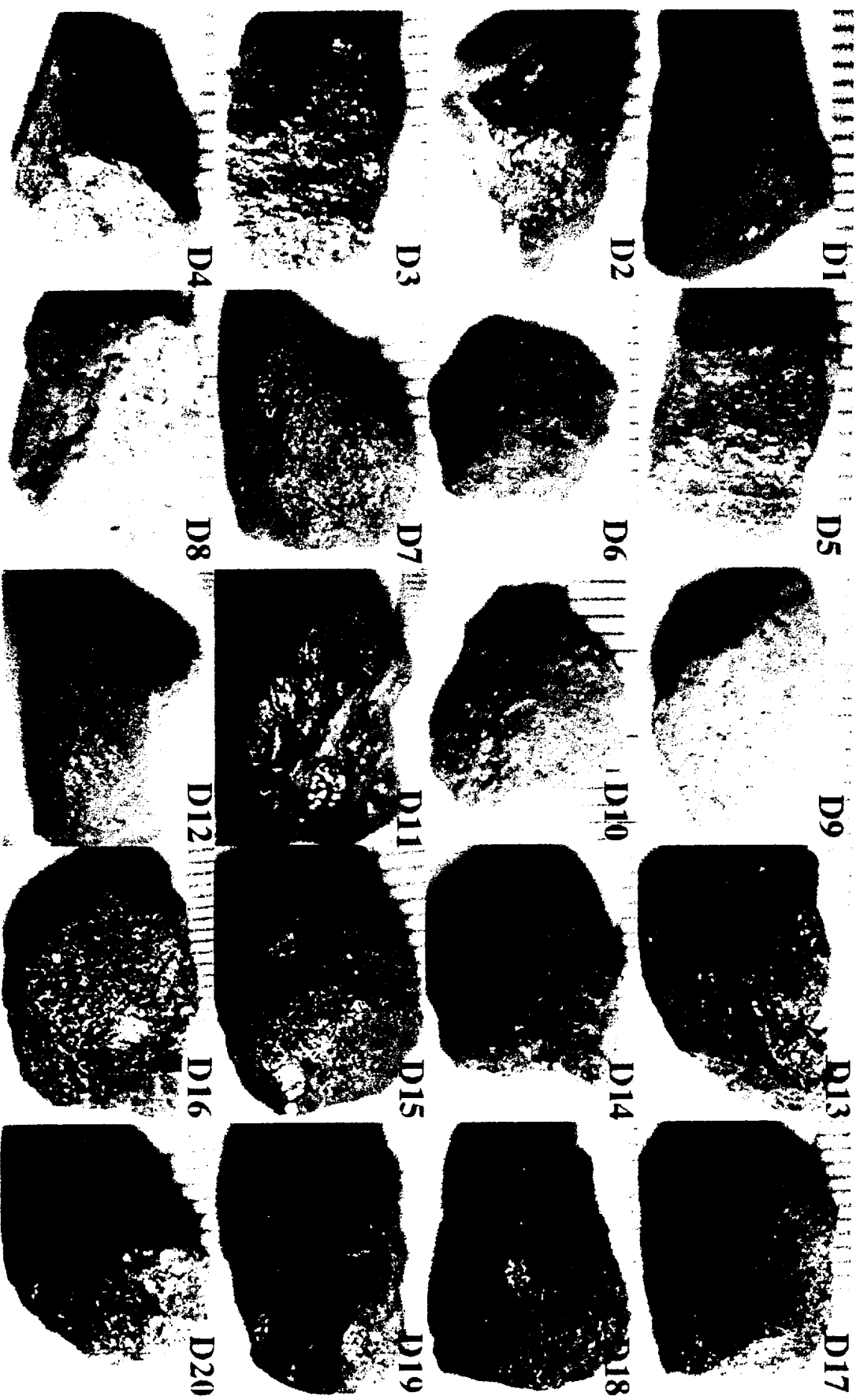
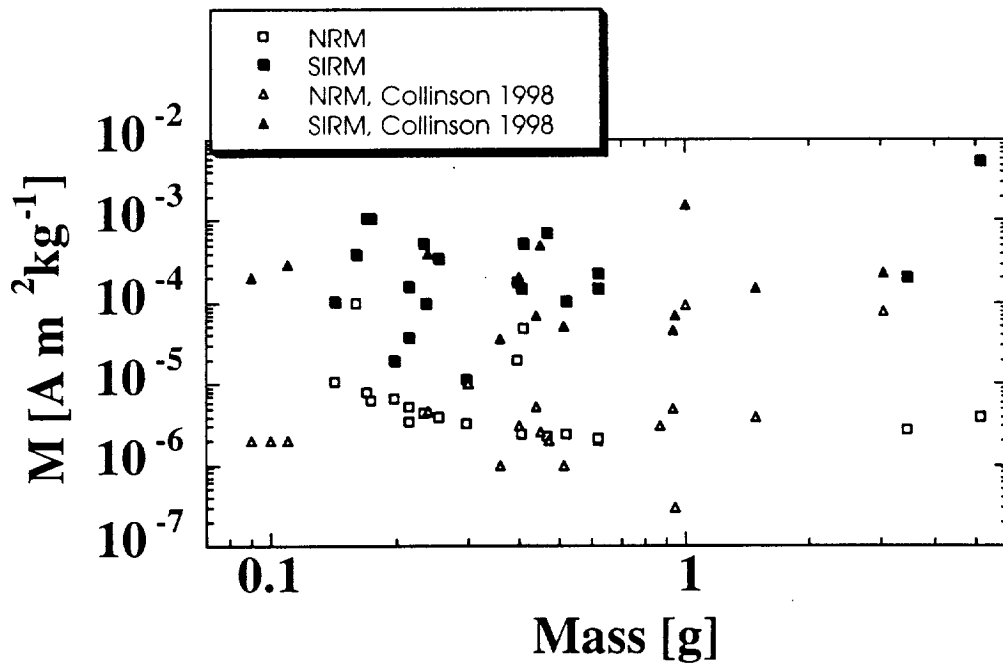
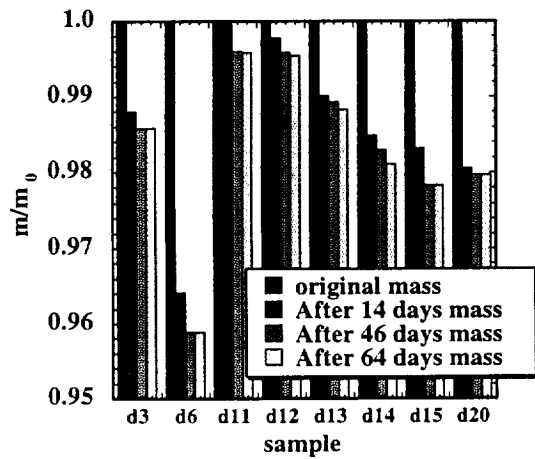


Figure 1:  
Morphology and sizes of carbonado diamonds from Central Africa. The scale is marked by distance between the lines and is 1 mm.

(a)



(b)



(c)

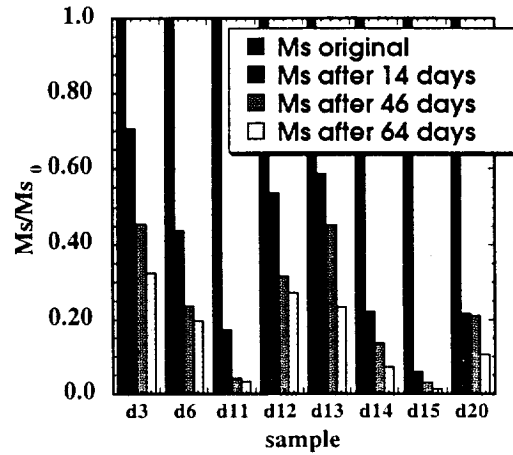
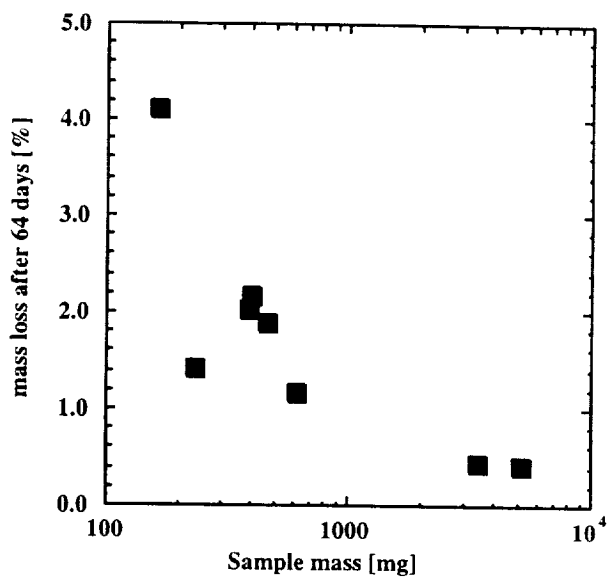
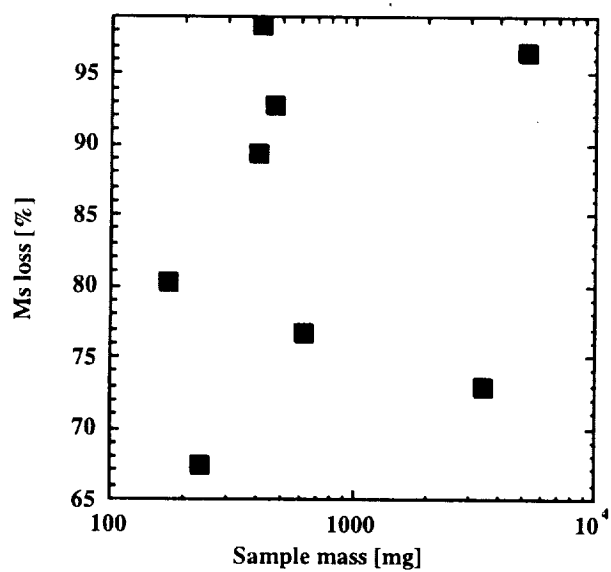


Figure 2:

(a) Remanent (NRM) and saturation remanent magnetization (SIRM) of carbonados from this and the Collinson (1998) studies. (b) Mass and (c) saturation magnetization loss due to acid treatment.



(a)

(b)

Figure 3:

Effect of acid treatment on magnetization (a) and mass (b) of the carbonado samples.

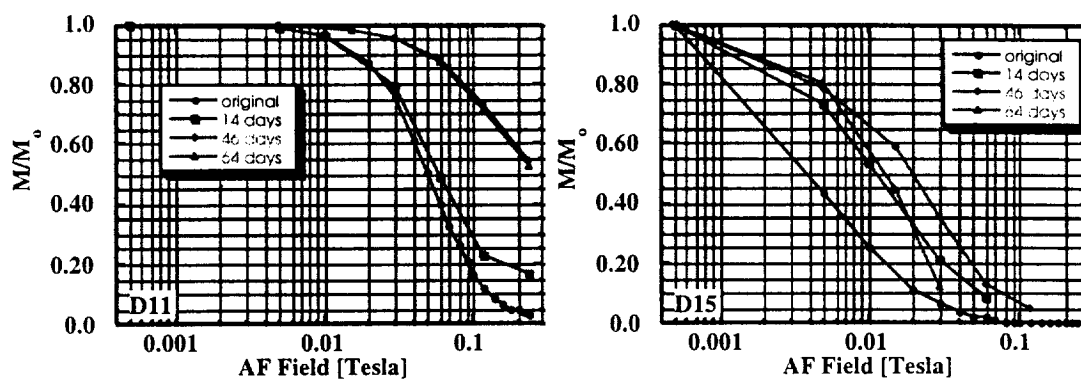


Figure 4:  
Hardening of magnetic grains in D11 and D15 as a result of acid treatment

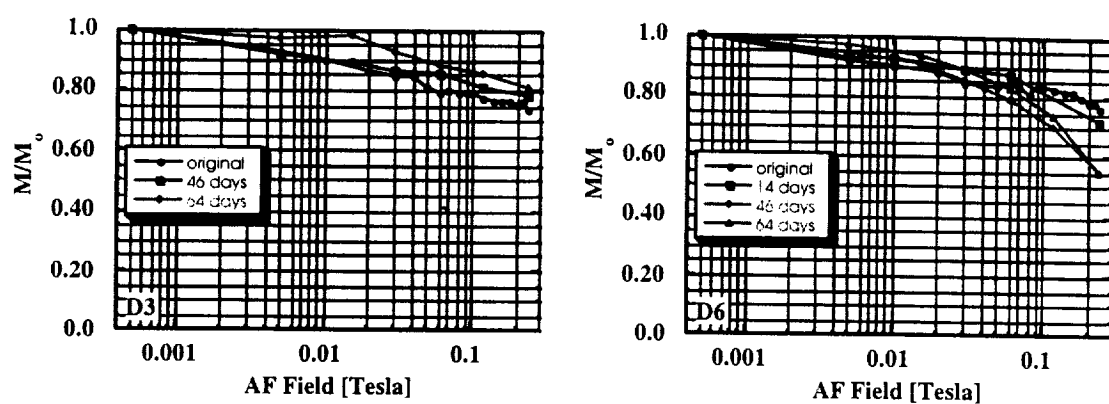


Figure 5:  
Acid treatment has very little effect on hardness of D3 and D6 carbonados.

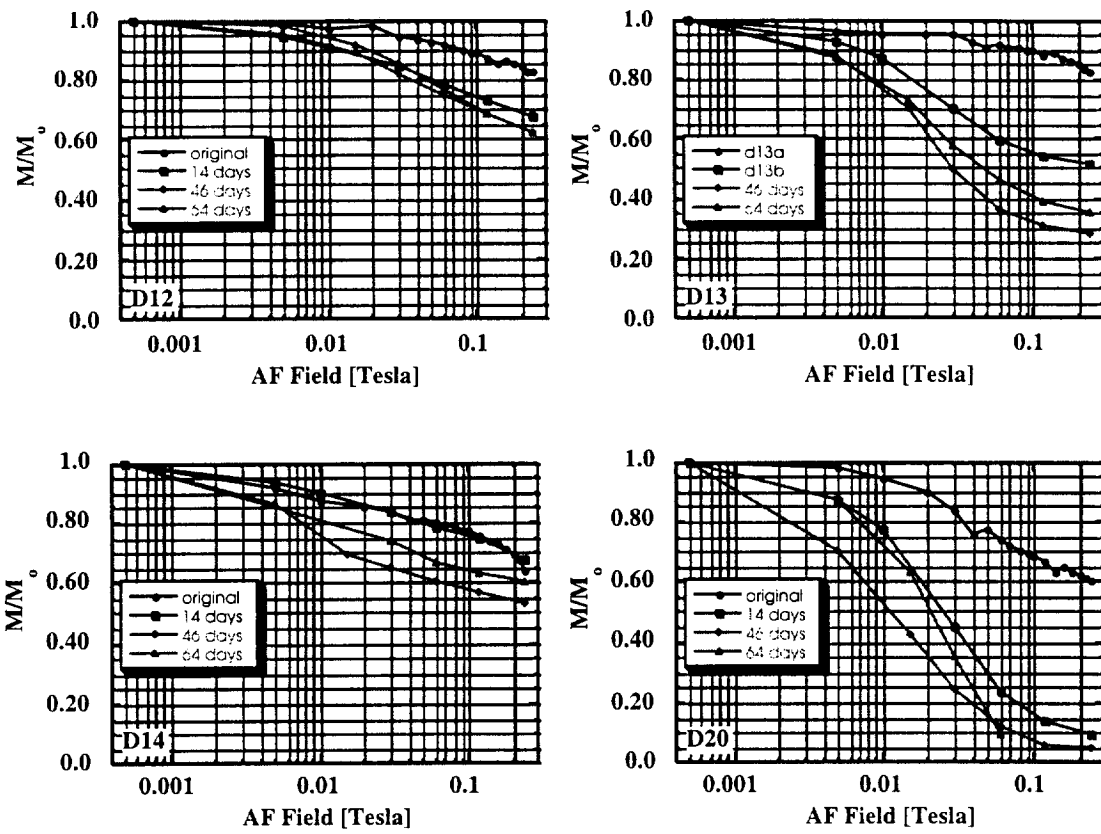
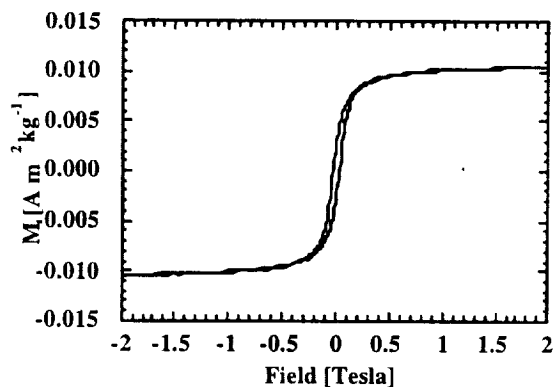
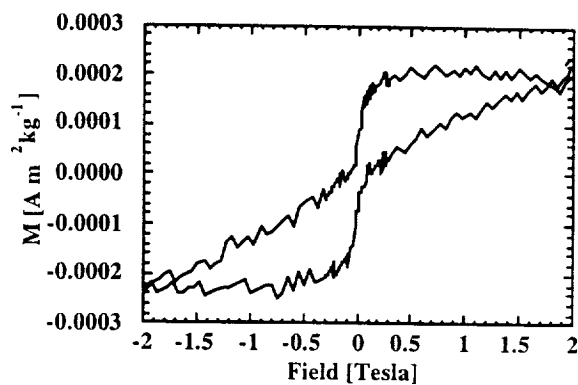


Figure 6:  
Acid treatment has softening effect on hardness of D12, D13, D14, and D20 carbonados.



A

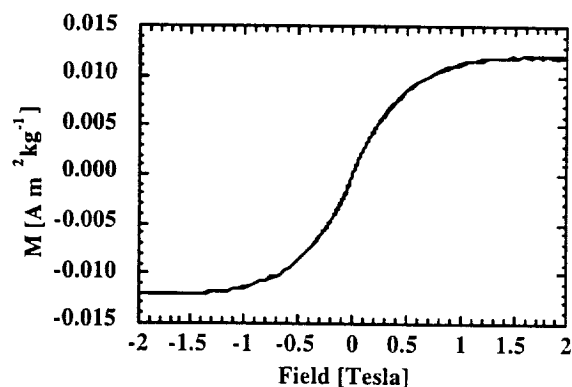
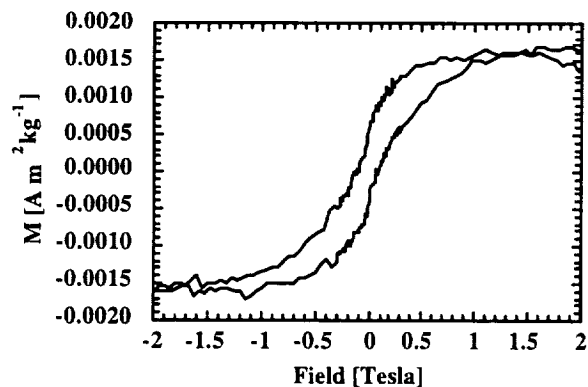


B

C

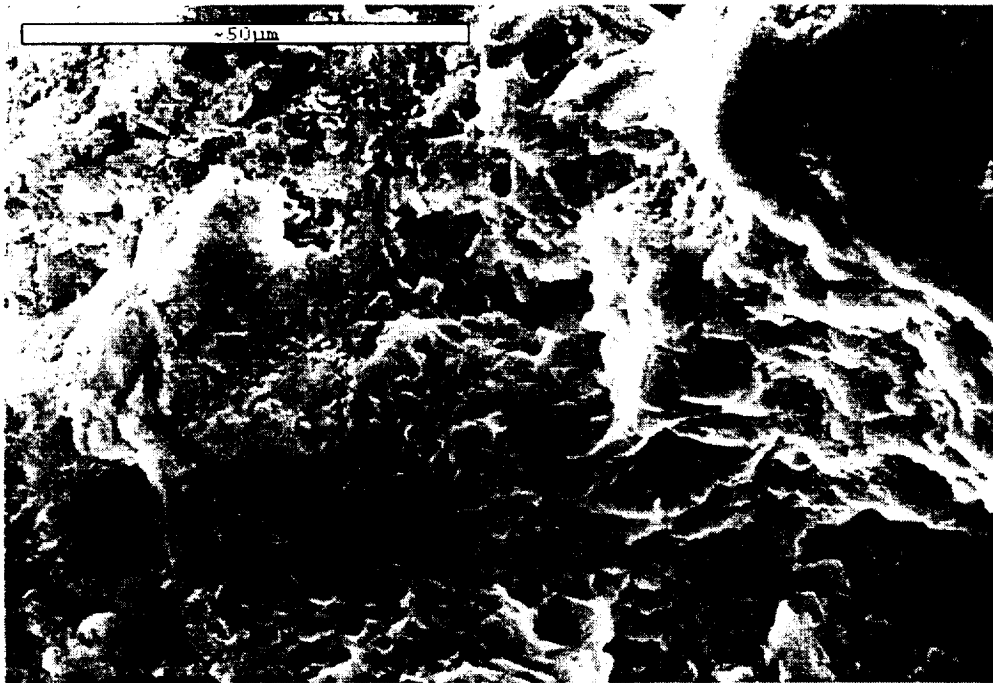
D

Figure 7:

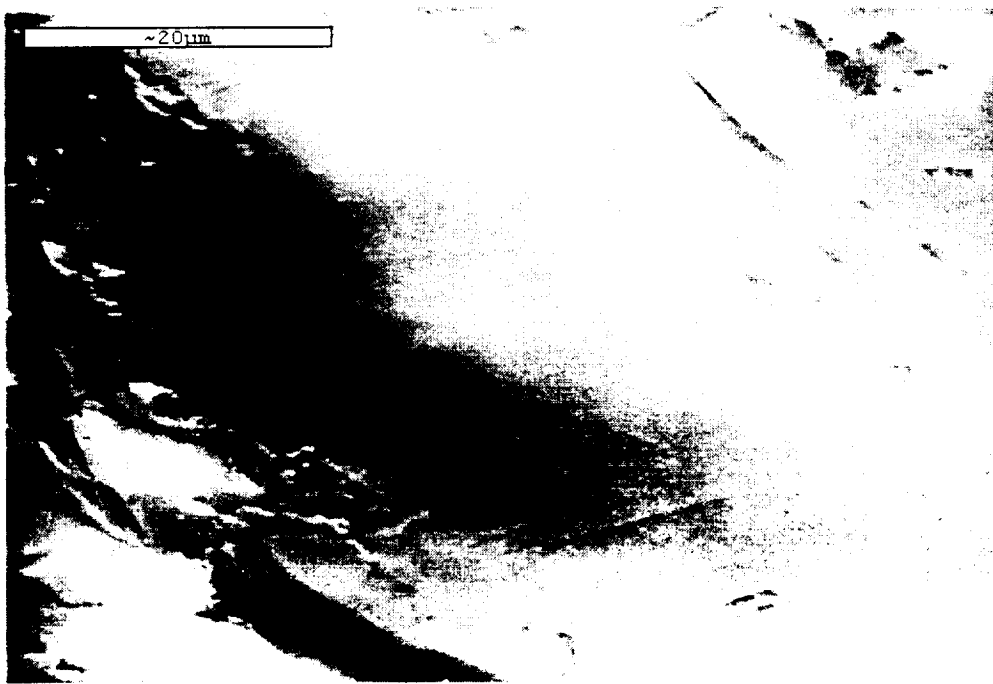


Hysteresis loops of carbonado diamonds. A. Sample D11, B. Sample D12, C. Sample D14, D. Sample D15. All loops were corrected for diamagnetic slope due to diamond host.



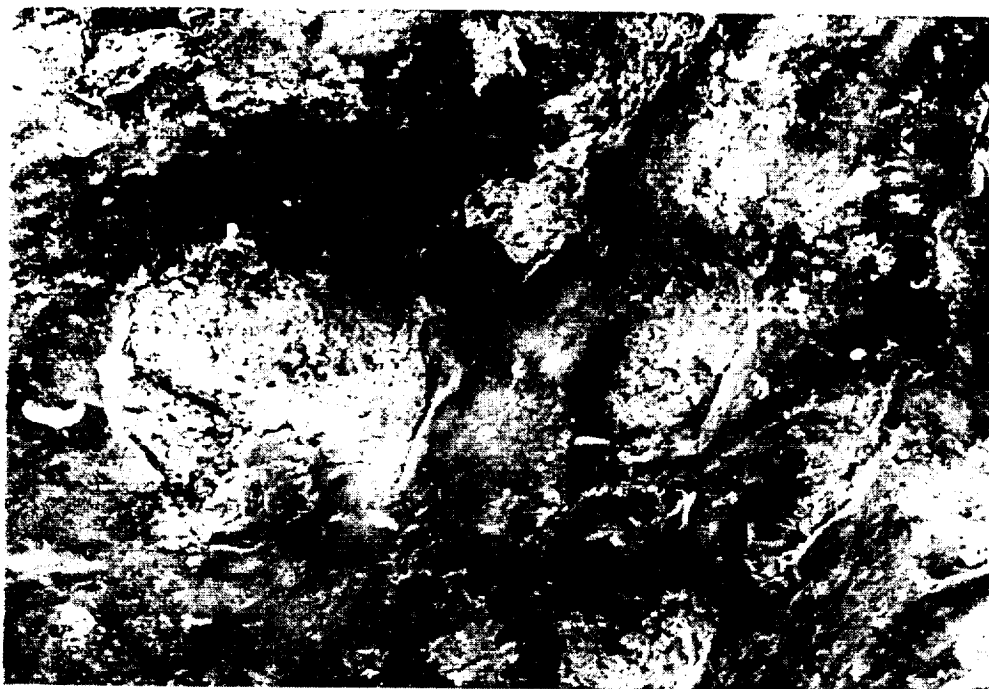


(a)

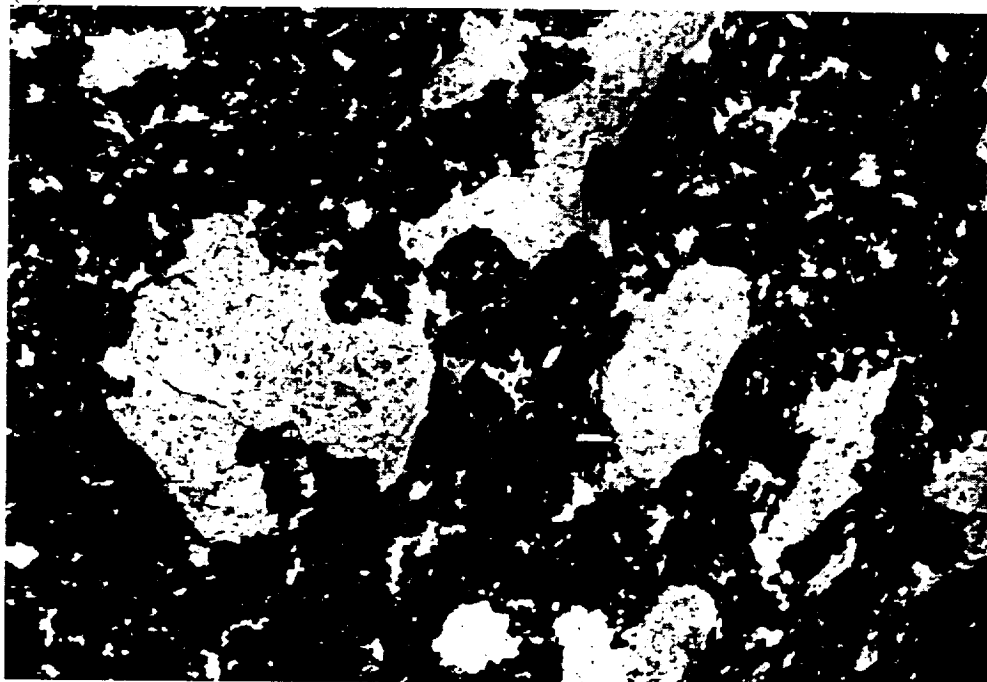


(b)

Figure 8: Modes of physical characteristic of the carbonado surface: (a) An internal surface of carbonado D19. (b) A smooth glassy carbonado surface (D1).



(a)



(b)

Figure 9: Example of the magnetic content concentrated within the pores of carbonado D11. The images are 50  $\mu\text{m}$  wide. (a) scanning electron image (b) back scattered electron image of the same area as (a) The bright pore fill is composed of iron oxides